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DESIGN AND CONSTRUCTION OF A
CHANNEL ELECTRON MULTIPLIER BASED
MÖSSBAUER SPECTROSCOPY SYSTEM

THESIS

Daniel J. Robbins

Captain, United State Air Force

AFIT/GNE/ENP/92M-9

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DEPARTMENT OF THE AIR FORCE
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DESIGN AND CONSTRUCTION OF A CHANNEL ELECTRON MULTIPLIER BASED MÖSSBAUER SPECTROSCOPY SYSTEM

THESIS

Presented to the Faculty of the School of Engineering

of the Air Force Institute of Technology

Air University

In Partial Fulfillment of the

Requirements of the Degree of

Master of Science in Nuclear Engineering

Daniel J. Robbins, BSNE

Captain, United States Air Force

March 1992

Approved for public release, distribution unlimited.

Preface

The purpose of this study was to design and build an improved detector system for the Air Force Institute of Technology's use in Mössbauer Spectroscopy research. While the apparatus has so far achieved only part of the goal established for it, I believe that only minor work is needed by a future student to make it an excellent laboratory instrument.

I owe thanks to many people for help on this project. Dr. George John, my thesis advisor, provided guidance, tutoring, and a verbal kick in the rear whenever I got too lazy. The model makers at the AFIT Fabrication Shop, Jack Tiffany, John Brauhaus, and the rest of the crew, were able to make anything I designed almost overnight, whether it made sense or not. Bob Knight of the 4950th Test Wing's Aeronautical Laboratory, provided much valuable information on high vacuum techniques, along with Captains Pete Halland and Jim Targrove of the AFIT Engineering Physics Faculty. Mike Ray, also at the 4950th TW lab helped with ceramic fabricating. Bill Hofele, at the fabrication unit for the 4950th TW, arranged for welding support and helped me find material. Bob Hendricks, Leroy Cannon, and Greg Smith, the lab technicians helped me find parts and put them together. Mike Blazejowski at Galileo Electro-Optics Corporation and Dr. Jeff Zabinski of the Air Force Wright Aeronautical Lab were

helpful with technical questions.

My understanding of Mössbauer spectroscopy was made clearer by three books not cited in the thesis: Lectures on the Mössbauer Effect, by J. Danon, published in New York by Gordon and Breach in 1968, Mössbauer Spectroscopy, edited by Dr. Uri Gonser, published in New York by Springer-Verlag in 1975, and Chemical Applications of Mössbauer Spectroscopy, edited by V. I. Goldanskii and R. H. Herber, published in New York by Academic Press in 1968.

Daniel J. Robbins

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Abstract

A channel electron multiplier (cem) detector system was designed and built to provide a signal to a Mössbauer spectroscopy system in an effort to obtain a better signal-to-noise ratio than achievable with a proportional counter system. Three cems are contained in a vacuum chamber built using standard components. Gamma rays of 14.4-keV energy from a ^{57}Co source enter from outside the chamber through a window and are absorbed by an ^{57}Fe target near the cems. The target emits internal conversion electrons which are collected by the cems and a Mössbauer spectrometer collects the signal. The system produces a usable signal, but the signal-to-noise ratio is unacceptably high. The strongest peak has a maximum count only 1.5 % of the total background count. The corresponding peak in a spectrum collected using the proportional detector system is 3.4 % of the total background count. One possible cause is that some gamma rays are striking two of the cems, thus producing ionizations in the detectors. The cems need to be repositioned. A screen should be added in front of the cems and grounded to reduce counting of low energy electrons. These improvements should reduce the background count rate.

DESIGN AND CONSTRUCTION OF A CHANNEL ELECTRON MULTIPLIER BASED MÖSSBAUER SPECTROMETRY SYSTEM

I. Introduction

The design of a laboratory apparatus requires the same consideration of economy, function, and usability as any engineering project. This thesis describes the design, construction, and testing of an apparatus for conducting Mössbauer spectrometry with conversion electrons from ^{57}Fe.

Background

The Air Force Institute of Technology (AFIT), in support of the Air Force Wright Laboratory (AFWL), is studying the corrosive effects of hot lubricants on iron. Researchers at AFWL use x-ray emission to study oxidation of iron on the surface of samples caused by exposure to hot lubricants. AFIT plans to use Mössbauer spectroscopy to study oxidation of the iron on the interior of the samples.

The Mössbauer effect is the non-doppler-broadened, recoil-free, resonance absorption of gamma rays. Free nuclei are in random motion, resulting in doppler broadening of resonant absorption energy widths. They also recoil to conserve momentum when they emit or absorb photons. If the nuclei are in a crystalline matrix they are not free to experience random motion and the solid as a whole shares the recoil energy. This results in elimination of the doppler

broadening and gamma emission and absorption without loss of energy. Resonant absorption occurs with the natural line width associated with the Heisenberg uncertainty relation centered on the energy of the photon.

AFIT currently uses a proportional counter to detect conversion electrons emitted by nuclei excited by the absorption of resonant gamma rays. Channel electron multipliers have a very low intrinsic background count rate. A system employing these detectors for conversion electrons will improve signal to background ratio.

Scope of Work

The goal of this study was to design and build a detection system using channel electron multipliers (cem) in Conversion Electron Mössbauer Spectrometry (CEMS) to produce a better signal to noise ratio than achievable with the proportional counter currently in use.

Work included system design, procurement or manufacture of parts, design modification as necessary, parts assembly, and system testing. Shortcomings of the system were noted and possible causes identified. Improvements were made where time permitted.

Chapter two of this thesis describes the design requirements, chapter three is a detailed description of the system, chapter four discusses system performance in testing, and chapter five gives conclusions and recommendations.

II. Design Requirements

As has been stated, the goal of this thesis is the design and construction of a system for using channel electron multipliers (cem) for Conversion Electron Mössbauer Spectrometry (CEMS). The important aspects in the design of the system are: (1) configuration, installation, and power requirements of the cems to obtain the maximum signal-to-noise ratio and (2) the design and construction of a high vacuum system which provides the conditions required for operating the cems in their optimal configuration. The design consideration involved in these two aspects are clearly coupled. For example, the optimal orientation of the cems, the target, and the excitation source will determine the design of the vacuum chamber. The final design must also take into consideration the materials that are available and the compromises needed to complete the system in the allotted time.

Signal Source

The source of gamma rays is ^{57}Co in a rhodium matrix. It decays by electron capture with a half-life of 271 days to an excited state of ^{57}Fe. Practically all, 99.98% (Lederer and Shirley, 1978:165), of the cobalt decays to the 136.5-keV level of ^{57}Fe (see Figure 1). The excited iron nuclei decay to the 14.4-keV level in 89.0% of the cases with the other 11.0 % decaying directly to the ground state. The 14.4-keV decay of ^{57}Fe produces internal conversion electrons as well as gamma photons so that 9.54% of all ^{57}Co decays result in

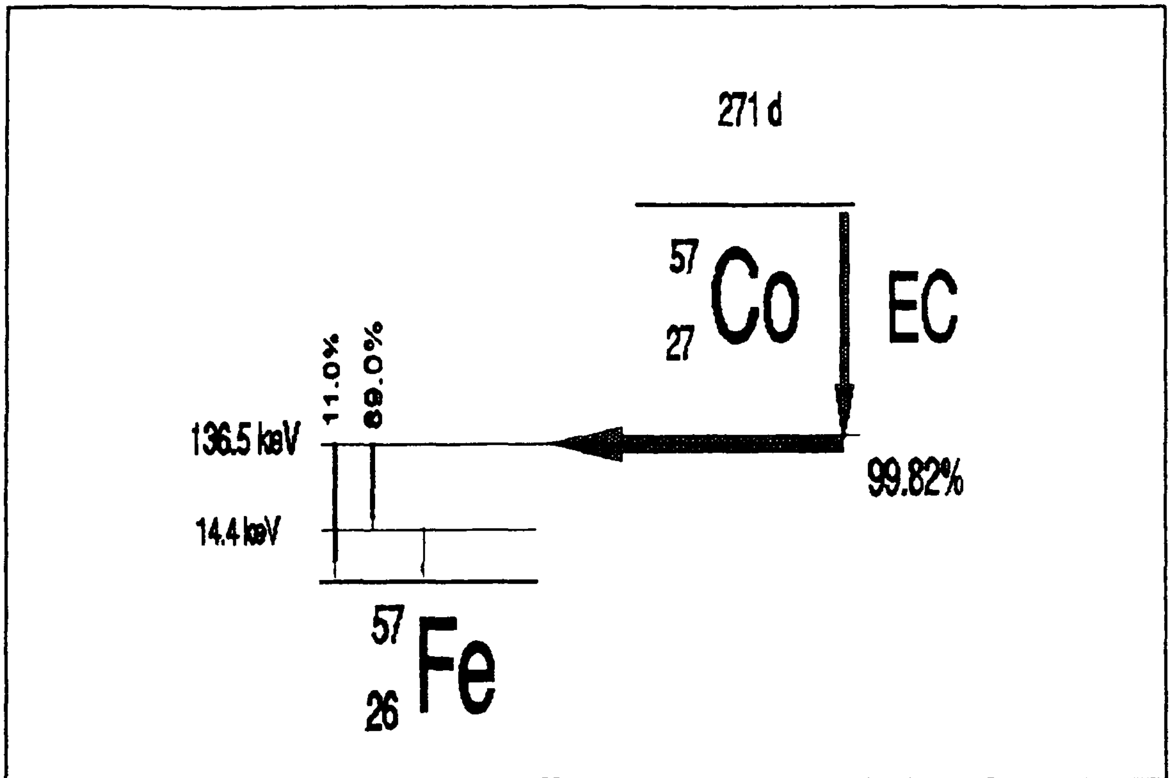

Figure 1 Decay scheme of ^{57}Fe, simplified to include only items of interest. Thick lines are ground state thin lines are excited states with energy level on left. From Lederer et al., modified to show only levels of interest.

14.4-keV gamma photons. The conversion electrons are easily absorbed in the media surrounding the iron/cobalt (the rhodium matrix), whereas the gamma photons are available to excite target ^{57}Fe nuclei through resonance absorption.

The target used in testing this system is 2000 Å of iron enriched in ^{57}Fe plated on a stainless steel substrate. When a gamma ray with the right energy strikes an ^{57}Fe nucleus it will excite to its first excited state, i.e. 14.4 keV. All nuclei excited to this state will decay to the ground state by emitting either internal conversion electrons (89.2%) or gamma rays (10.8%, see Table I). The source and target radiations

Table I Important gamma ray and conversion electron intensities per decay of ^{57}Co (^{57}Fe). Excerpted from NCRP Report #58.

Radiation	Energy (keV)	Intensity (%)
γ_1	14.4127	9.54 ± 0.14
γ_2	122.0614	85.6 ± 0.7
γ_3	136.4743	10.58 ± 0.08
ce-K-1	7.3007	69.6 ± 0.5
ce-L-1	13.5666	7.79 ± 0.22
ce-MNO-1	14.3198	1.15 ± 0.07

drive the signal-to-noise ratio considerations.

The signal strength is limited by the number of conversion electrons available for counting. At best, only 9.54% the source decays produce gamma rays which can excite the target nuclei. Of the gamma rays absorbed by target nuclei, only 89.2% will produce internal conversion electrons. This limits the conversion electrons from the 14.4-keV resonance absorption to 8.5% of the source intensity. Meanwhile, the gamma rays not absorbed by the target nuclei produce background counts.

Gamma rays are highly penetrating. The gamma rays from the source are isotropically distributed in emission direction so that many will interact in the region of the detectors with material other than the target nuclei and produce background counts. A detector system is needed which is efficient at collecting and counting the small number of conversion electrons from the 14.4-keV decays in target and is not

strongly affected by the scattered or direct gamma rays. Cems are good prospects for this application.

<u>Channel Electron Multipliers</u>

Channel electron multipliers (cem) operate in a manner similar to a photomultiplier tube. (Knoll, 1989:262-263) They are shaped like a funnel attached to a tube (see Figure 2). An electric potential is applied between the funnel and the opposite end of the tube. Electrons that enter the funnel scatter into the tube and are accelerated by the potential. As each electron strikes the wall of the tube secondary electrons are ejected which cascade down the tube to produce up to 10^8 electrons at the end of the tube where they are collected to produce a signal (Henkel and Gray, no date:122).

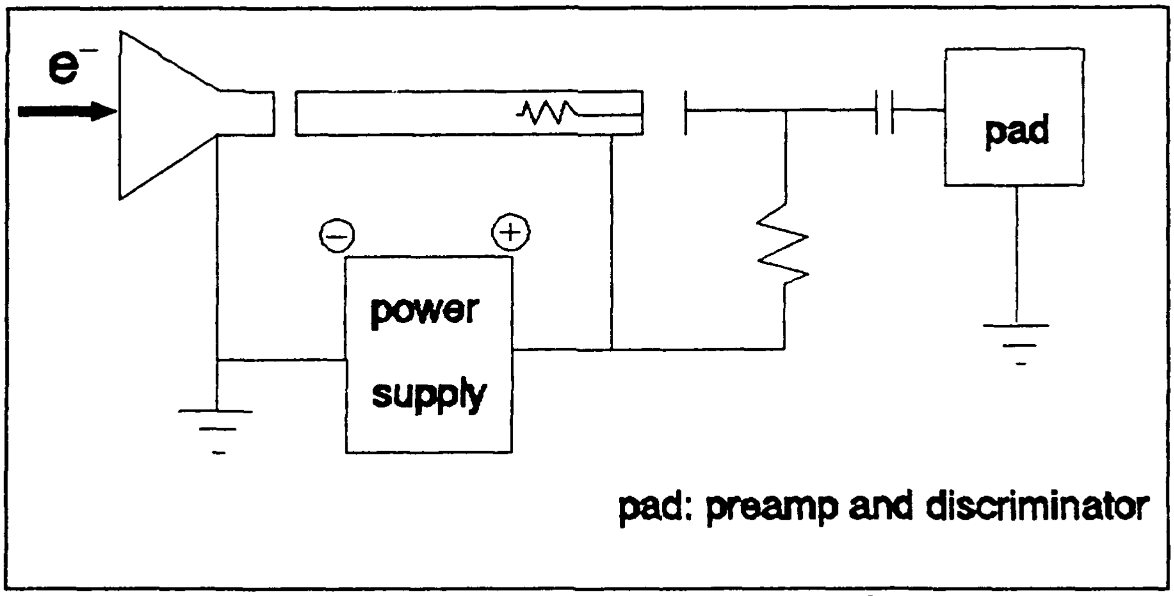

Figure 2 Schematic of channel electron multiplier. From Galileo Electro-Optics Corporation product brochure.

Cems have three attributes which are important for this system. They are strongly direction biased, i. e. only electrons which enter the funnel will be counted, they have a

strong multiplication factor so that a weak input produces a usable output signal, and they have a low intrinsic background count so that a weak signal can still produce a good signal to noise ratio.

Cems must be operated in pressures below 10^{-6} Torr to protect the multiplying coating and to prevent electrical discharge from the high potential needed for operation. The lower the pressure the longer will be the life of the cems and the lower will be the number of ionizations in the air about the target and the cems. Lower pressures also mean fewer molecules between the target and the detectors to attenuate electrons. In addition, lower pressure allow gasses to desorb from the sensitive coating of the cems. This reduces the production of positive ions in the cems and the consequent back-streaming of these ions after pulsing.

Cems are also sensitive to contamination. Trace amounts of contaminants, especially hydrocarbons, on the surface of the cem can also result in arcing or burning in the region of the contamination. The vacuum system must protect the cems, as well as provide the low pressure for their operation.

High Vacuum

Four goals were established for the design of the vacuum system. First, an operating pressure of 10^{-8} Torr was selected because it was well below the maximum operating limit for the cems but high enough to be achieved in a reasonable pumping time without baking. Second, the system had to be oil free to avoid contaminating the cems. Third, since Mössbauer spec-

trometry is sensitive to relative motions, the system had to be vibration free. Fourth, the system needed to be compact to fit in the available space in the laboratory. The high vacuum itself produced two more concerns, out-gassing and virtual leaks.

In high vacuum many materials will out-gas. Components or adsorbed molecules that would remain in the material at normal pressures have sufficiently high vapor pressure to break free in high vacuum. Rubber, some plastics, and some metals, e.g. zinc or alloys containing zinc and stainless steels with a high carbon or high sulfur content, should be avoided (O'Hanlon, 1989:279-298).

Tight spaces, threaded surfaces and surfaces in macroscopic contact but not bonded, result in virtual leaks. At atmospheric pressure molecules of air will be forced into the microscopic gaps in these joints. As the pressure drops to extremely low levels some molecules remain in the spaces but since the walls are close together the molecules can not flow freely out. The pressures are low enough that the molecules are collisionless and act independently of one another. The molecules tend to bounce back and forth between the walls of the threads or joints. The molecules in these spaces require a long time to diffuse into the vacuum chamber so that they can be pumped out of the system.

Material Limitations

Two constraints affect engineers in any enterprise, time and money. While no explicit limit was placed on funds which could be spent on this project, it was required that the project be completed for the minimum amount of money. To allow time for testing the system, overcoming problems, correcting faults and optimizing performance, assembly had to be completed as soon as possible. Whatever was to be done had to be completed within three months in order to allow the thesis to be written.

Both constraints require that wherever possible parts, machinery, and materials already on hand be used. If parts needed to be ordered they were to be off-the-shelf to avoid the expense and time of special order fabrications. Where nonstandard items were required first consideration was given to modifying standard parts, especially those already available at AFIT or another Wright-Patterson AFB unit.

III. System Description

The system is compact, mobile, rugged, and contained within a single platform except for the Ranger Scientific MS-1200 Mössbauer spectrometer. Here is a full description.

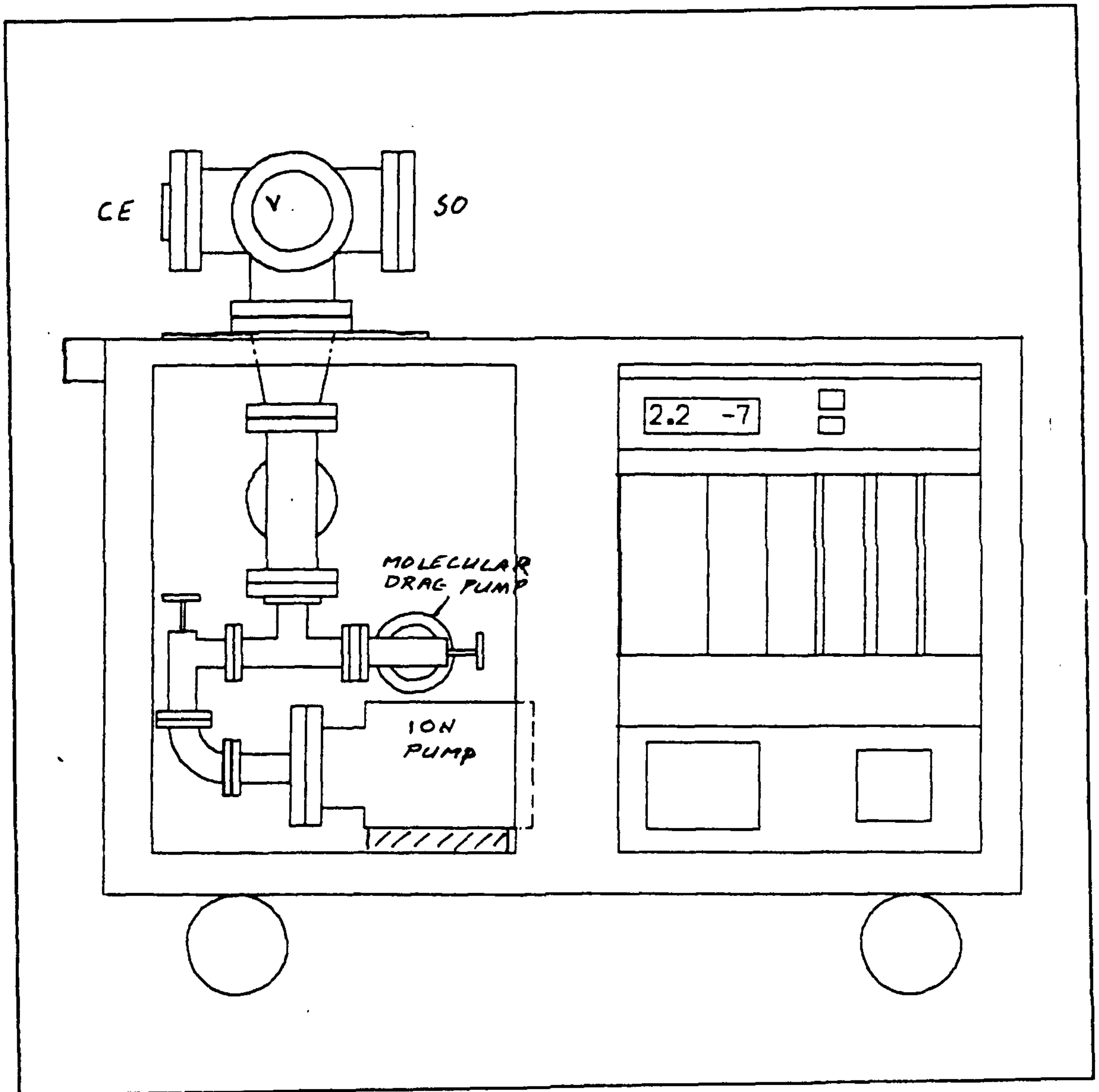

Figure 3 CEM detector and vacuum system.

Vacuum System

In house or standard off-the-shelf parts are used. All flanges are Conflat (CF) compatible except the one on the

molecular drag pump which is ISO standard. All references to stainless steel should be understood to mean 304 or 304L stainless steel unless specifically excepted.

An equipment cart was identified which could serve to house the system (see Figure 3). At 24 inches wide by 46 inches long by 34 inches high, it is compact and is mounted on wheels to permit movement of the system. It has a reliable brake to increase stability when the Mössbauer system is operating. The long dimension is divided into two equal bays, each with equipment mounting racks of standard size. It also incorporates a power strip with grounded outlets. One bay houses the vacuum chamber and pumps while pump and gauge controllers and a NIM bin are mounted in the other bay. The vacuum system was designed to fit this cart.

A standard 5-way cross was selected to be the primary chamber because it was the largest size on hand. It consists of five stainless steel tubes welded together at right angles to form an intersection as shown in Figure 4. The outside diameter (OD) of all tubes is 4.000 inches with a 0.083 inch wall thickness to allow an inside diameter of 3.834 inches. This diameter dictated the size of the assembly on which the cems were to be mounted. This also determined the size of the re-entrant well which would contain the window for entry of the 14.4-keV photons from the Mössbauer source and the collimator/shield. The cross formed by four of the legs is mounted horizontally with the fifth leg projecting downward.

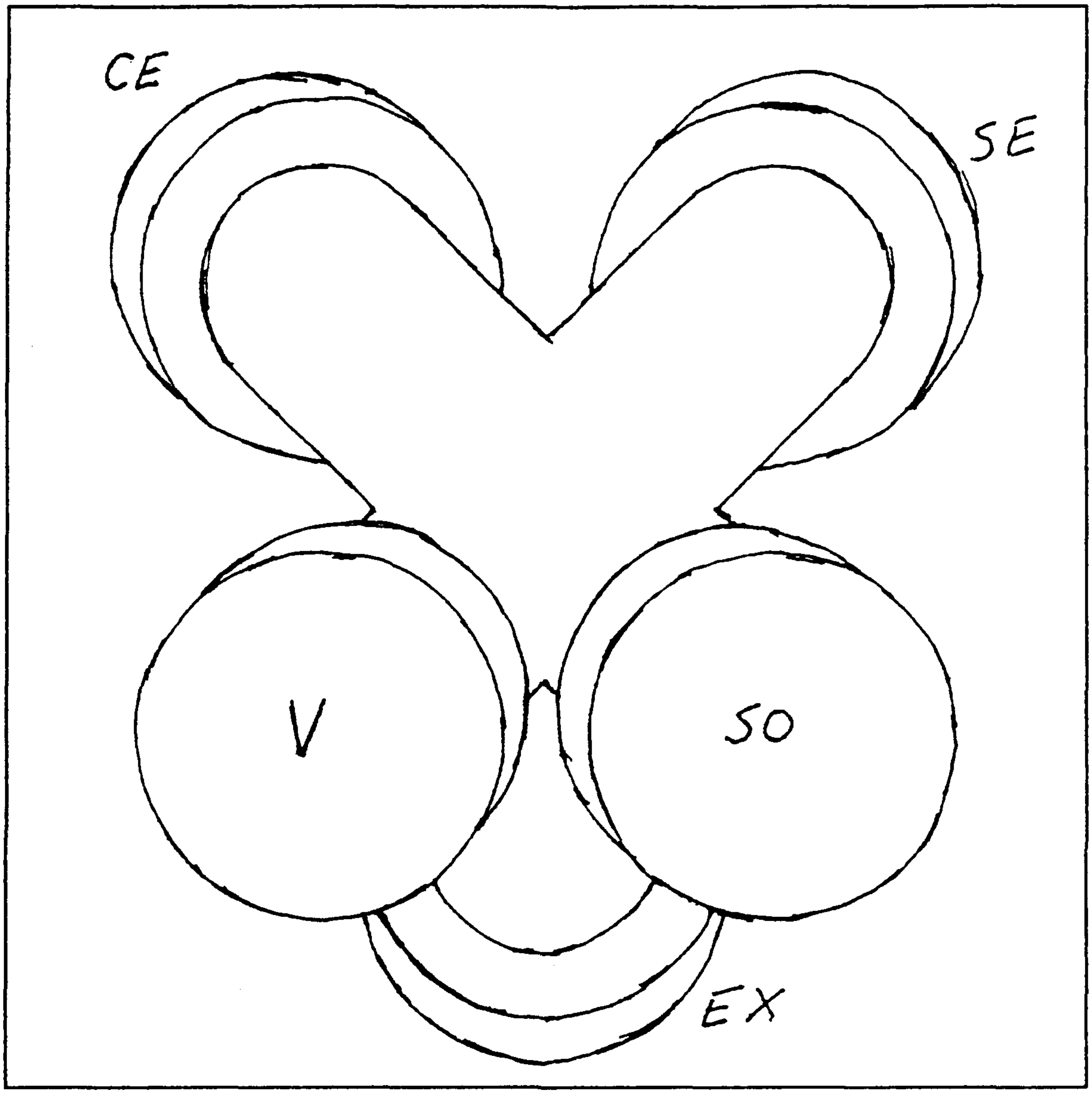

Figure 4 5-way cross which forms primary chamber of vacuum system.

All of the legs, labeled CE, SO, SB, V, and EX in Figure 4, are fitted with 6-inch CF flanges, three are rotatable and the other two are fixed. Leg CE is used to emplace the cems and the target, it has a rotatable flange. The opposite leg, SO, contains a re-entrant well with a 25 mil PBT window for entry of gamma rays from the source and has a fixed flange. The leg labeled SB is used to supply bias power to the cems

and collect the signal from the cems. A 6-inch to 2.75-inch zero-length reducer flange is attached to the rotatable flange on this leg. The feed-through for bias voltage and signal are MHV coaxial connectors on a 2.75-inch CF flange. The leg labeled V is empty and has a view port mounted to the fixed flange on the end to allow observation. The final leg, EX, is used for connection to the pumps and gauges through a conical reducer nipple and has a rotatable flange.

The conical reducer nipple passes through an eight inch square hole cut in the top of the equipment cart over the center of the bay housing the vacuum system. Two 1/4 inch thick rectangular aluminum plates close around the reducer nipple. The plates are 7 X 14 inches and cut to close around the reducer nipple. The flange of the reducer nipple rests on the plates to support the vacuum system. The aluminum plates are held in place by four 1/4 inch diameter bolts slipped through holes drilled through the outside corners of the plates and into the top of the cart. This arrangement facilitated the construction of the vacuum system by allowing sub-assemblies to be built prior to mounting in the cart and permitting position adjustments prior to final assembly. The narrow end of the conical reducer nipple is connected to one leg of a Tee with 4.000-inch CF flanges.

One leg of the Tee projects horizontally to the rear of the equipment rack and is used to mount a tubulated ion gauge. The leg of the Tee opposite the conical reducer nipple connects to the middle leg of a 2.75-inch Tee through a zero

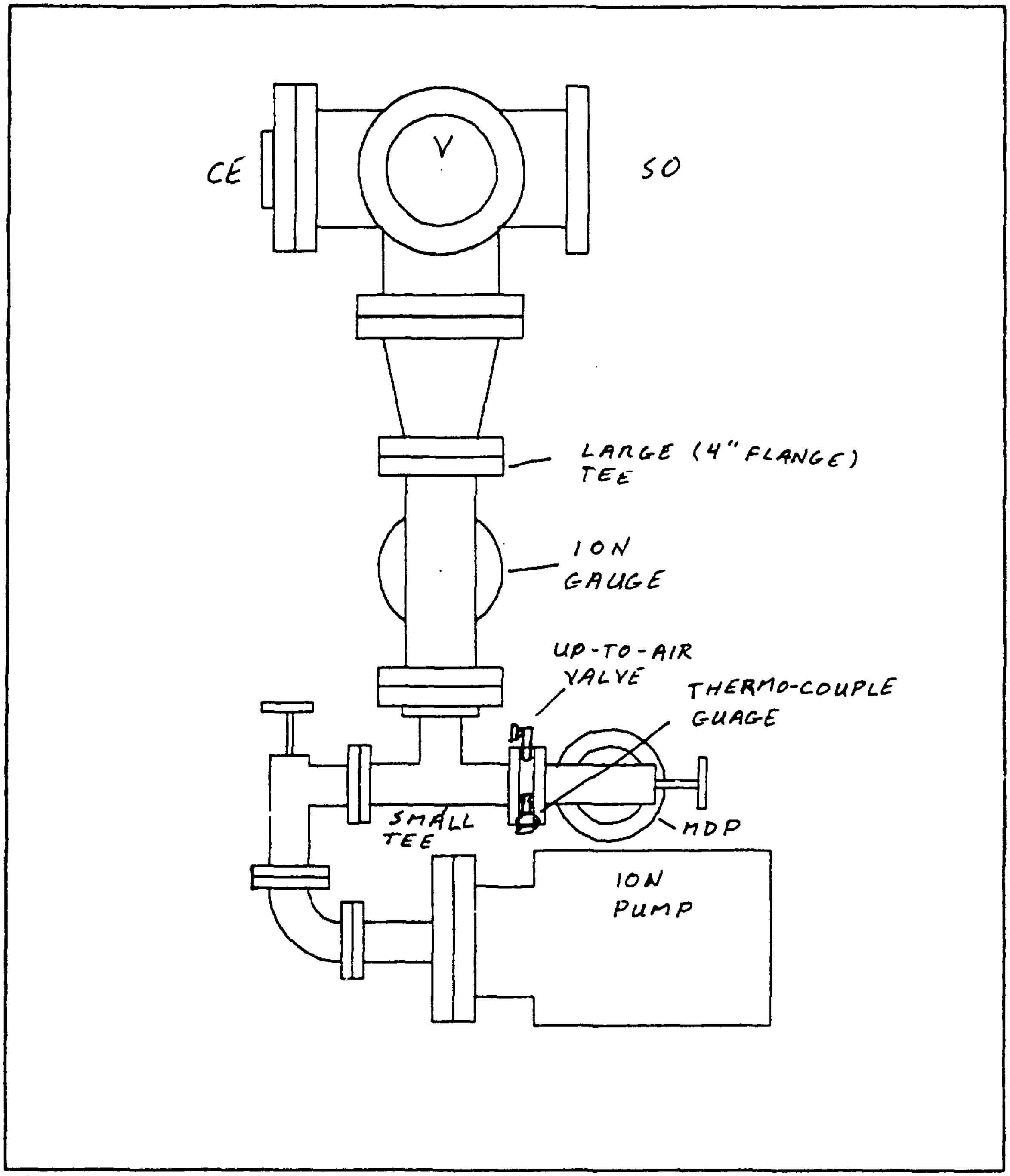

Figure 5 Vacuum system, frontal view without cart.

length reducer flange, shown in Figure 5.

The other two legs of the Tee with 2.75 inch CF flanges follow the long axis of the cart and are connected to hand operated angle valves. One valve opens to a vacuum ion pump

through a 90-degree radius elbow and a 6-inch to 2.75 inch reducer nipple and the other valve opens on a molecular drag pump through a Conflat to ISO flange adapter nipple. The outlet of the drag pump is connected to a diaphragm pump by heavy duty vacuum hose.

A flange housing a thermocouple vacuum gauge and up-to-air valve is mounted on the primary chamber side of the drag pump valve, between the valve flange and the 2.75-inch CF flange on the Tee. This arrangement allows the chamber to be brought to atmospheric pressure with the valves to all pumps closed. It also allows an inert gas, i.e. nitrogen, to be fed to the inlet of the up-to-air valve to reduce recontamination of the system by moist room air when brought back to normal pressure after the initial pump down.

Pumps

Three pumps are used to bring the vacuum system to desired pressure, a diaphragm pump, a molecular drag pump, and a sputter ion pump. The diaphragm pump produces a modest vacuum suitable for operating the molecular drag pump. The molecular drag pump achieves a much higher vacuum, necessary for starting the sputter ion pump. The sputter ion pump can achieve a higher vacuum than the molecular drag pump at a high pumping rate and has no moving parts.

Diaphragm Pump

In this system the roughing pump is a KNF Neuberger Incorporated model UN 725.3 diaphragm pump. Performance data are not available. Two flexible diaphragms draw air through

the inlet and force it out into the ambient air through one-way valves. The inlet is connected by 1/2 inch inside diameter heavy duty vacuum hose to the outlet of the molecular drag pump. It is effective in reducing the pressure of the vacuum system from atmospheric to a few Torr.

The molecular drag pump allows air to pass through it while it is turned off, so that the diaphragm pump may operate independently. However, the two pumps ordinarily operate together.

<u>Molecular Drag Pump</u>

The molecular drag pump is an Alcatel model 5010. It's basically a high speed turbine designed to operate with low inlet and exhaust pressures. A motor rotates a drum at a high velocity, 27,000 rpm in the Alcatel 5010. This produces a drag force which moves molecules from a region of low pressure to one of higher pressure (Barrington, 1963:38-39). The minimum inlet pressure depends on the exhaust pressure. With exhaust pressure reduced to a few Torr by the diaphragm pump the drag pump can bring the vacuum system down to the order of 10^{-6} Torr. To get to lower pressures the sputter ion pump takes over.

<u>Sputter Ion Pump</u>

In a sputter ion pump a high potential, on the order of 10^3 volts, is raised between electrodes made of chemically active material called a "getter" (Barrington, 1963:93-94). The potential accelerates the electrons and ions. A strong magnetic field, on the order of 10^3 gauss, traps the electrons

so that there are more collisions and additional ionization of the gas. On colliding with the wall some ions are embedded in the wall or react chemically with the getter. Some of the getter molecules are also forced into the gas through collisions. There they combine with gas molecules to form low vapor pressure compounds and condense onto the walls.

In this system a Varian Vacion model 911-5034 sputter ion pump is used. While it can function from 10^{-2} to 10^{-11} Torr it extends the pump life greatly if it is not started above 10^{-4} Torr. The volume pumping rate is approximately constant volume rate at 60 liters/second. It requires no lubricants which could contaminate the cems and since there are no moving parts there is no vibration to interfere with the velocity calibration of the transducer motor.

<u>Valves</u>

The system uses three valves, a hand operated angle valve between the primary chamber and each of the pumping channels, and a needle and seat type up-to-air valve with a port directly to the primary chamber.

The angle valves consist of 1.5-inch outside diameter tubes with 2.75-inch CF flanges forming a right angle. A screw post passes through the angle and is centered on one leg. The post is capped by the valve seat and is surrounded by a stainless steel baffle to seal the valve mechanism from the vacuum.

One angle valve has a gold seal, the seat is a knife edge which makes metal to metal contact with the moving seat,

cutting into the surface. This valve is used between the primary chamber and the ion pump with the seat port facing the pump so that the baffle will be in the chamber with higher pressure.

Another gold seal valve was not available, so the other valve has a Viton O-ring seal. This is used between the primary chamber and the drag pump with the seat port on the primary chamber side. Again, the baffle is placed in the higher pressure side of the valve.

The valve for bleeding in air uses a metal needle and a metal seat. The metal seat cuts into the needle to make a tight seal.

That is the system that achieves and maintains the high vacuum. The next section discusses the components housed in the vacuum chamber.

Source Re-entrant Chamber

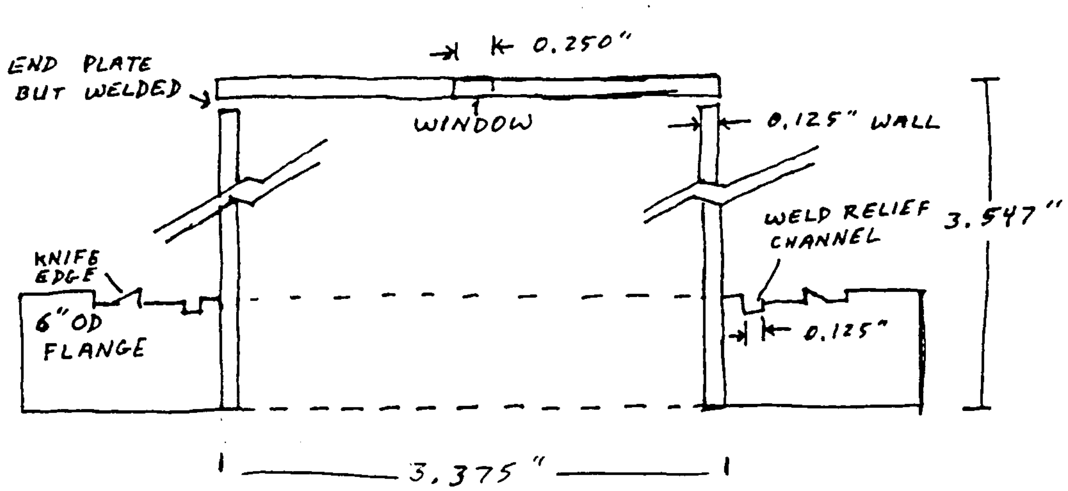

Figure 6 Vacuum chamber re-entrant well.

The transducer motor must remain outside the vacuum chamber. Its size, electrical wiring, and moving parts would greatly complicate the vacuum chamber environment. The chamber wall could not pierced for the shaft on which the source is mounted and maintain a high vacuum seal. The gamma rays would be significantly attenuated if they had to penetrate the stainless steel chamber walls to reach the target and the source needed to be as close to the target as possible to get a better signal by putting the most intense gamma flux possible over a maximum target area. The solution to these problems was a re-entrant well in one leg of the 5-way cross.

Components for the re-entrant well, which contains the entry window for gamma rays and the lead collimator and shield, are shown in Figure 6. The components were machined from stock stainless steel to give a wall thickness of 0.125

inches and are TIG welded on the vacuum side of the well. The PBT window, which is 25 mils thick, is fastened on the atmospheric side of the well with high-vacuum (low vapor pressure) epoxy.

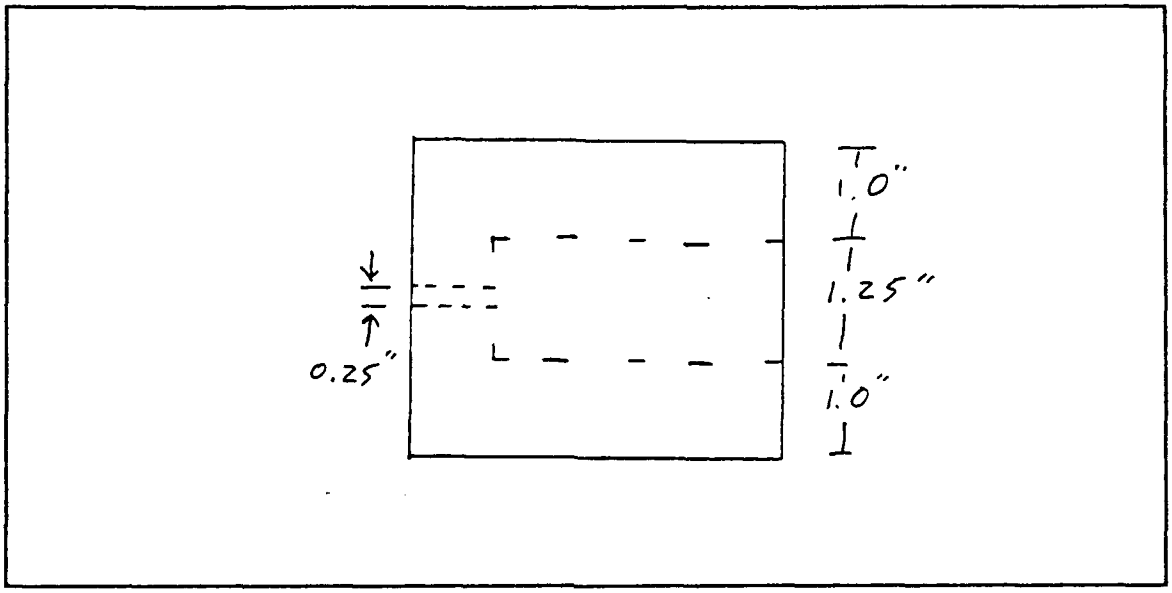

Figure 7 Collimator and shield.

A lead cylinder, shown in Figure 7, was cast and machined to fit the re-entrant well. A tunnel approximately 1 inch in diameter was drilled down the center of the lead cylinder to 1.0 inch from the end. The last 1.0 inch was drilled 1/4 inch in diameter in line with the window in the re-entrant. The source, mounted on the transducer motor shaft, fits in the tunnel so that the lead cylinder shields the source from the laboratory and collimates the beam in the direction of the target. When mounted in the 5-way cross the re-entrant/collimator/shield extends approximately 1/8 inch into the intersection.

Detector Assembly

The detector assembly is the most complex part of the system. The primary components are the cems. The other components are mechanical support, an electric field shroud, and electrical connections.

Channel Electron Multipliers

The cems selected for this system are model 4820 Channeltrons manufactured by Galileo Electro-Optics Corporation. They have funnel openings 1.0 inch in diameter and are about two inches long from the funnel opening to the anode. The tube is curved to form a coil to increase the probability of electrons striking the walls of the tube, thereby increasing the gain. As tested by Galileo, each of the Channeltrons has a gain on the order of 10^8 and a dark count of zero counts for sixty seconds operating with a bias of approximately 2400 volts. Three cems were mounted together to form a single detector.

The cems are arranged in a triangle, shown in Figure 8, facing the target location. The planes of the funnel openings form a rough tetrahedron with the surface of target. This maximizes the area of coverage while leaving a gap about 3/8 inch in diameter through the middle of the cems to allow unhindered passage of the gamma beam. Numbered I, II, and III on the support platform for identification, they are connected electrically in parallel to create the equivalent of one detector with a large collection area.

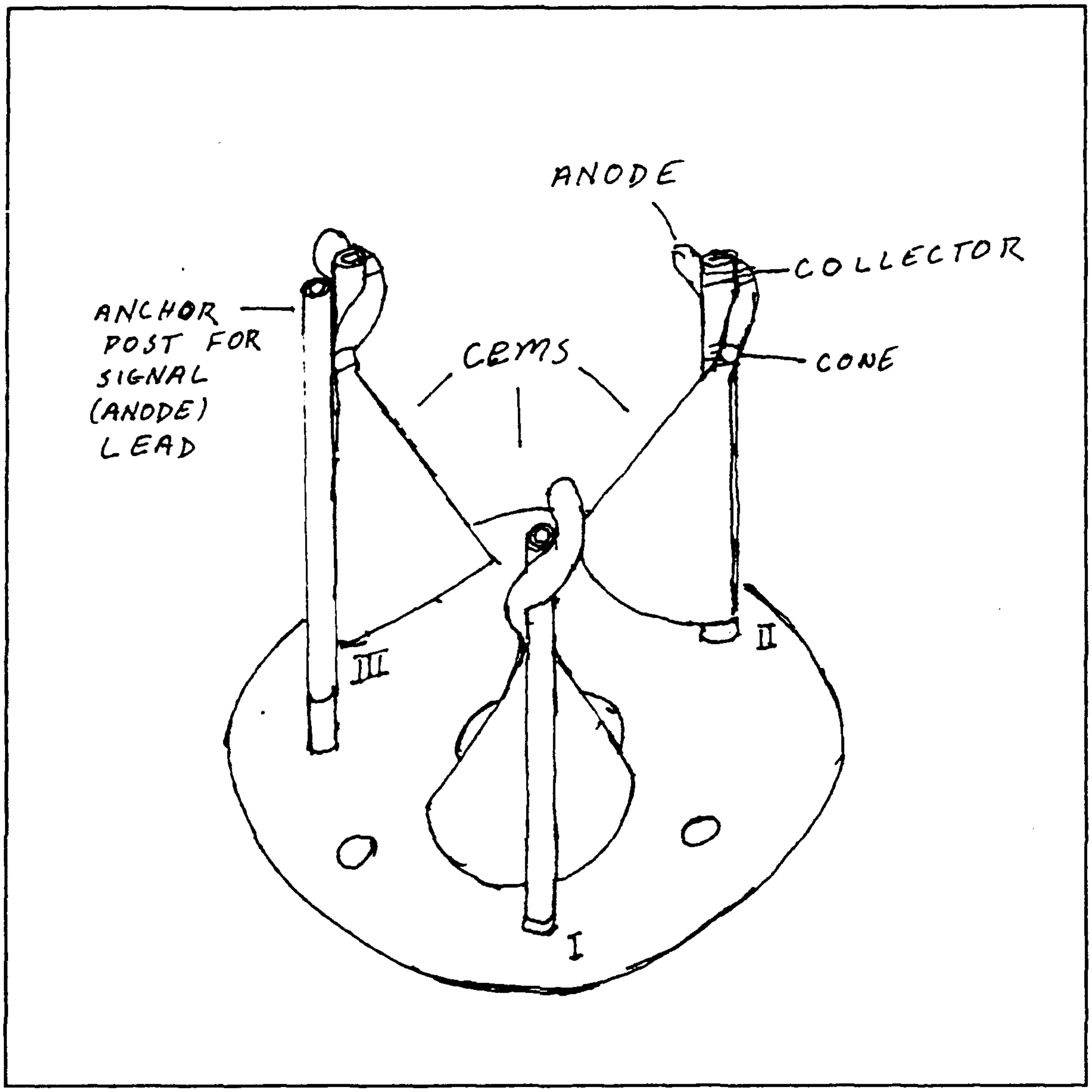

Figure 8 CEM detector assembly with electric field shroud removed.

Mechanical Support

A support platform for the cems, seen at the bottom of Figure 8, was fabricated from oxygen free copper. The platform is 1.850 inches in radius and has a 1.00 inch diameter hole in the center. Copper was chosen because it is a nonferrous conductor. A conductor was desired to form part of an electric field isolation around the cems and a non-

ferrous material was desired to avoid possible background interference with the ^{57}Fe target. Oxygen free copper was chosen for its low out-gassing in a vacuum. The cems are attached to this plate through alumina posts.

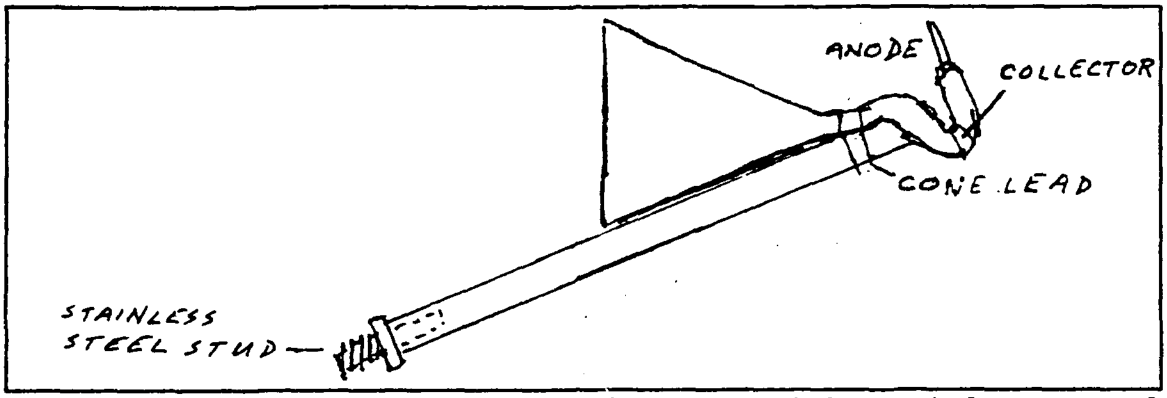

Figure 9 Cem mounted on ceramic post with stainless steel stud.

Each cem is mounted on an alumina (aluminum oxide, Al_2O_3) tube 3 inches long and approximately 3/16 inch in outside diameter with a wall thickness of approximately 1/32 inch thickness, as shown in Figure 9. Alumina was chosen because it is a rigid insulator with low porosity so that it won't out-gas contaminants into the vacuum (Beck, 1964:Vol 3, 9-11). Mounting was accomplished by attaching two nickel bands to each post using a low vapor pressure epoxy suitable for high vacuum and spot welding the input and output electrodes of the cems to the nickel bands. The cems are mounted with the edge of the cone parallel to the ceramic post. This places the center line of the cems at a 22.5 degree angle from perpendicular to the plane of the target. The center of the cem funnel openings are approximately 1.0 inch from the support platform.

The posts are attached to the support platform with stainless steel studs.

Stainless steel studs 1/2-inch long with integral 1/4-inch long machine screws were machined to slip fit inside the cem support posts and were epoxied in place. Three slotted holes 1/2 inch in length were cut radially into the copper plate at 120 degree intervals and centered 1.514 inches from the center of the plate. Two flats were cut in the studs's machine screws to facilitate escape of trapped gasses. The screws fit through the slotted holes of the support platform and are held in place by stainless steel, type unknown, nuts and star lock washers. The posts, hence the cems, can be adjusted slightly, inward or outward along the support plate radius. It was desired that the cems's positions be adjustable lengthwise along the mounting posts and in angle, but the articulations necessary for such adjustments would create an assembly too large for the 5-way cross. Time and budget did not allow finding a larger primary vacuum chamber, so the limited adjustability of the cems was accepted. An additional ceramic post with stainless steel mounting stud was made and is mounted on the plate in a fixed position to support the signal leads from the cems. The mounting plate is supported by a 6-inch to 2.75-inch zero-length reducer flange attached to the five-way cross.

To attach the cem support plate to a 6-inch CF reducer flange, three 1/4-inch diameter stainless steel posts were sunk into holes 1/4-inch deep drilled into the flange

1.375 inches from the center at 120 degree intervals. The stainless steel posts are 3.5 inches long. The last inch of each post is threaded with relief slots cut in the threads to allow out-gassing. Holes matching the positions of the steel posts are drilled in the cem support platform. The posts slip through the platform and nuts are placed on either side to hold it in place. The platform is adjustable over a range of about 1/2 inch by adjusting the nuts.

After assembly, it was found that the platform and cems did not extend as far into the chamber as desired. Extensions were made which thread onto the end of the posts supporting the platform. The platform slips over studs on these extensions and is held in place by nuts. The extensions can then be adjusted to properly position the support platform. Relief channels for out-gassing are cut into the studs on the extensions.

Electric Field Shroud

An electric field shroud was designed to enclose the cem assembly. This shroud consists of the cem support platform, a copper cylinder, and a copper top. It is electrically isolated from the cems and grounded to the vacuum chamber through the support platform mounting posts. It is intended to contain the electric field of the cems.

The wall of the shroud was made by forming a cylinder approximately 3.5 inches long and 3.7 inches in diameter from 0.016 inch thick copper plate. The edges of the plate were overlapped and joined together with stainless steel screws.

Many 1/4 inch diameter holes were punched in the cylinder to allow freer flow of gasses through the chamber. The cylinder slips over the cems and is attached to the irons on the support platform by stainless steel set screws.

A cover for the open end of the cylinder was made from the same copper sheet. The cover has four tabs which are attached to the cylinder by stainless steel set screws. A 1/2-inch diameter hole is cut in the center of the top to permit passage of the gamma ray beam. When the detector assembly is in position in the vacuum chamber, the top of the electric field shroud is about 1/8 inch from the wall of the re-entrant well.

<u>Electrical Connections</u>

Each cem has three leads for electrical connections, one for the inlet or cone, one for the collector or tail, and one for the signal. The leads of the three cems were combined in parallel. Four inch nickel strips were attached to the three master leads and passed through the electric field shroud. Nickel was chosen because it is a flexible conductor with good vacuum behavior and it can be attached by spot welding. This allows solders, which have poor vacuum behavior, to be avoided. The signal lead was anchored on the fourth ceramic post, added for that purpose. The other two leads were anchored on cem support posts.

<u>Electrical Feed Through</u>

An electrical feed-through manufactured by MDC with four MHV connectors is used to pass the bias voltage and signal

into the vacuum chamber. One connector is used for each of the cem leads, leaving one connector unused. The feed-through is on a 2.75-inch CF flange which bolts to a 6-inch to 2.75-inch zero-length reducer flange which in turn bolts to a flange on the 5-way cross.

The nickel leads from the cem assembly are fastened to the coaxial connectors with sleeves were machined from oxygen free copper. Set screws in the sleeves pinch the leads against the pins. The leads are prevented from unintentional grounds by teflon sleeves. Teflon was chosen because it's a good insulator, is unabsorbent, and has a low vapor pressure (Beck, 1964:Vol 3,220-221).

Voltage Divider

To provide a potential to each of the three leads from a single high voltage power supply, a voltage divider box was built (see Figure 10). This allows a positive potential on the cones of the cems to increase the collection of electrons and a large potential between the cones and the collectors of the cems to accelerate electrons.

A potential of a few hundred volts is desired at the cone while a potential difference of 2500 to 3000 volts is needed between the cone and the collector. Two 2.2 megohm resistors are placed in series between the collector output and the cone output. An 800 kiloohm resistor is put between the cone output and ground. A simple application of Kirchoff's laws shows that the ratio of the voltage at the collector output to the cone output will be 6.5 : 1 so that a power supply output

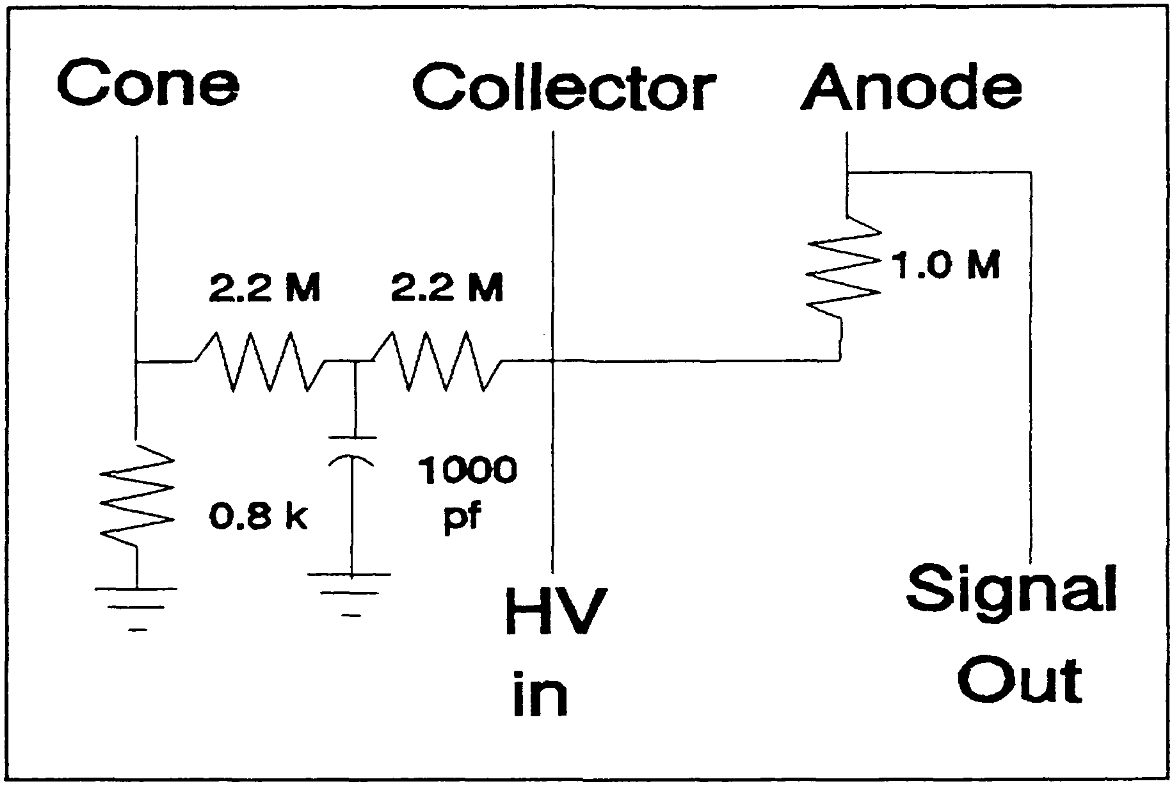

Figure 10 Schematic of voltage divider box.

of 2600 volts will produce 2600 volts at the collector and 400 volts at the cone for a bias of 2200 volts on the cem. The capacitor between the 2.2 megohm resistors helps keep the potential constant as current draw increases with increasing count rate.

Target Support

The target is supported on a 2.75-inch CF flange by a telescoping arrangement of stainless steel tube and shaft, shown in Figure 9. A stainless steel tube of 3/8-inch OD, 1/4-inch ID, and 3.0 inches long was set in a hole drilled 1/4 deep in the center of the flange and TIG welded in place. The last 1/2 inch of the free end of the tube was threaded, four slots were cut the length of the threads, and the tip of the

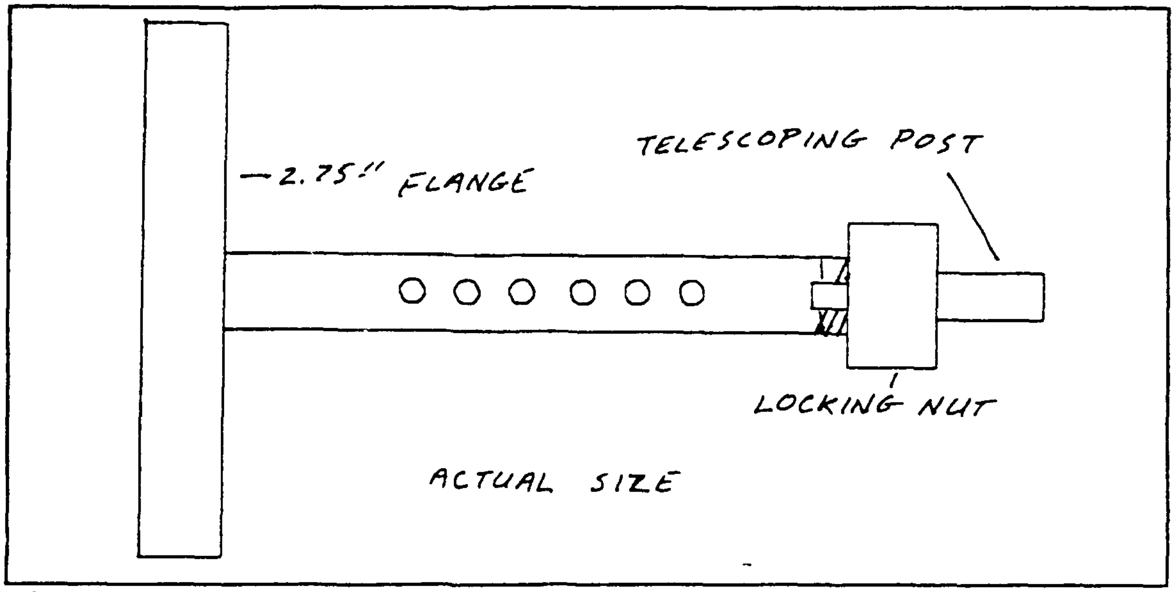

Figure 11 Target support on 2.75-inch CF flange.

tube was beveled. The slots allow out-gassing from the threads and allow the tube to flex. Several small holes were drilled along the length of the tube to allow gas to escape the tube interior. A knurled knob with a bevel matching the one on the tube was machined from stainless steel to fit the threads. When the bevels come in contact the tube is squeezed inward, forming a clamp.

Two 3.5 inch long shafts, 1/4 inch in diameter, were machined from stainless steel. The shafts slide into the tube and are clamped in place with the knob. The exterior end of one shaft is cut at a 90 degree angle while the other is cut at 45 degrees. This allows the target to be placed in two different positions. Because the intensity of electrons from a Mössbauer spectrometry target is known to vary with angle to the target plane, it was foreseen that in some circumstances

it might become desirable to use only one cem with the target plane angled towards the cem in use.

For testing the system the target was held in place by a copper wire. Short copper wires were epoxied to the ends of the shafts. The target used in testing has a hole in the center which was passed over the wire and the wire bent to hold the target in place.

For future use two containers were machined from oxygen free copper. The container base can be epoxied to the target support shaft. The cap of the container will then fit over the target and base, held in place by a pin. These containers were not finished and attached to the shafts because the intended targets have not been available.

For testing the system, the shaft with the 90 degree end cut was scored with a file. The marks were cut at roughly 1/4 inch intervals in the last inch of the shaft. They provide reference points for positioning the target with respect to the cems.

Gauges

There are two pressure gauges in the system. A Granville-Phillips model 274008 tubulated ion gauge is mounted to one leg of the Tee section with 4-inch flanges and a Hastings-Raydist model CVT-15A thermocouple vacuum gauge is attached to the flange containing the up-to-air valve. The ion pump also serves as a gauge of pressure through its current draw.

Instrumentation

Most instrumentation is contained in the remaining bay of the equipment cart. There is the ion pump controller, a Varian VacIon Pump Control Unit model 921-0062, and the Alcatel model CFV 10 molecular drag pump controller. Above these is a NIM bin with several pieces of equipment manufactured by EG&G Ortec, the high-voltage power supply, a single-channel analyzer, an amplifier, a timer/counter, and a pulse generator. This equipment was included to allow testing the cem system performance independent of the MS-1200 spectrometer. The ion gauge controller, a Granville - Phillips model 330, is mounted above the NIM bins.

The voltage divider, the MS-1200 preamp, model P-1200, and the controller/meter for the thermocouple pressure gauge rest on the top of the cart.

Mössbauer Spectrometer

A Ranger Scientific model MS-1200 Mössbauer spectrometer is used to vary the source gamma energy and collect the spectrum. To produce the small variation in gamma energy at the target, the source is mounted on the shaft of a transducer motor. The motor is connected to a controller which also accepts input from the detectors monitoring the source through a matching preamp. The controller relates the signal from the detectors to the velocity of the target and sends the data to a microcomputer for processing. The computer analyzes the data and produces a spectrum by plotting counts against the source velocity.

IV. System Performance

This section discusses system performance. After a brief description of testing method, the performance of the two major parts of the system, vacuum chamber and detectors, is summarized. Overall, the system produces a signal which the MS-1200 system can use to collect a spectrum, but the signal to noise ratio is not acceptable. Some of the possible causes have been identified and are soluble.

Testing Method

The approach to testing the system was simple and straight forward. After construction was completed, an attempt was made to achieve and maintain high vacuum. When the pressure was low enough for safe operation, below 10^{-6} Torr, power was supplied to the cems.

The first step was to initialize the cems by running a "clean-up" on them. The cems were given a potential of 2600 volts and a ^{90}Sr source (212 mCi) was placed in the re-entrant well. This operation was performed for several hours to minimize after-pulsing of the cems. Once after-pulsing was eliminated attempts were made to collect a spectrum.

Test spectra were collected with 2500 volts supplied to the cems. The source, 37 mCi of ^{57}Co, mounted on the transducer motor shaft, was placed in the re-entrant well. While spectra data were being collected, gross counts were measured by running a parallel lead from the spectrometer to the counter/timer.

Vacuum

An adequate vacuum was attained with only minor problems. The system pumps down to 10^{-5} Torr in a few minutes, 10^{-6} Torr in a few hours, and 3.5 X 10^{-7} Torr overnight. The system reaches the minimum pressure attained of about 5 X 10^{-8} Torr after two to three days of pumping. It slowly drifts between 4 X 10^{-8} and 6 X 10^{-8} Torr, but goes no lower. This is acceptable performance. The cems can operate with normal life expectancy at 3.5 X 10^{-7} Torr so that 24 hours or less after pumping is started counting can begin.

Detectors

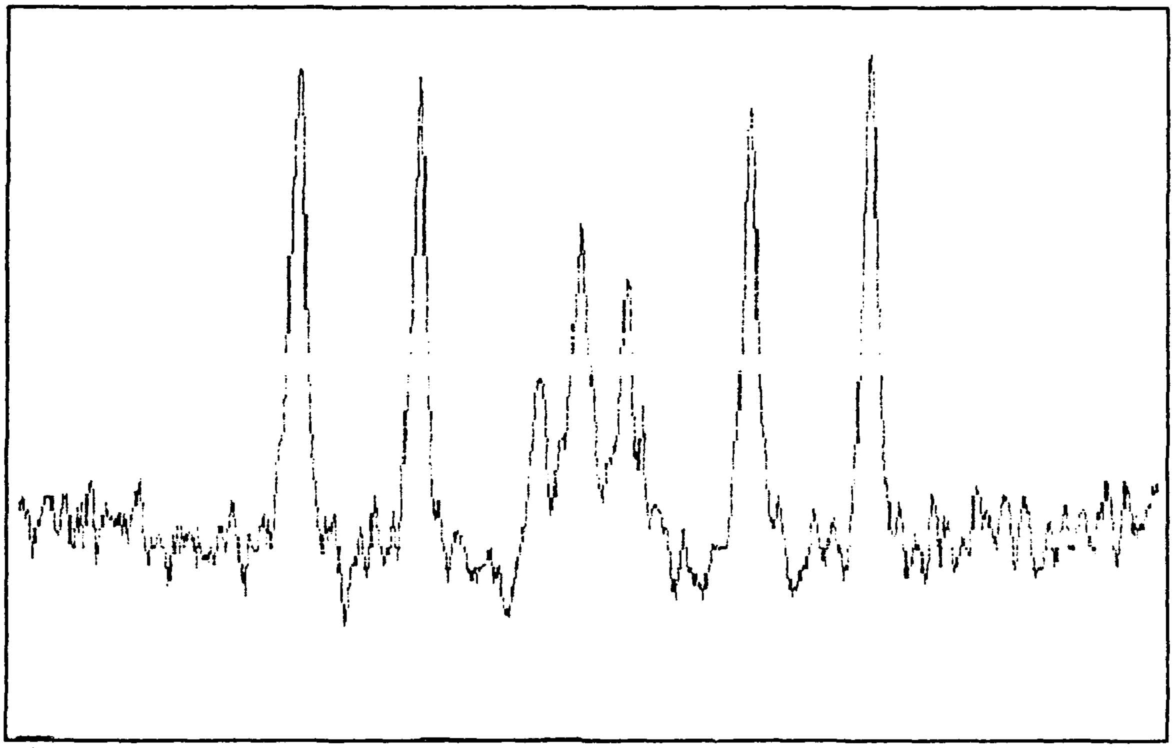

Figure 12 Mössbauer spectrum collected using proportional counter. Obtained in two days with 2000 Å of ^{57}Fe on stainless steel foil.

For comparison to the cem system, a spectrum, shown in Figure 12, had been obtained using the same source and target

with a proportional counter system. Counts were collected for about 48 hours. The parabolic curve results from the modulation of the distance between the source and target. The intensity of gamma radiation on the target falls off by the square of the distance between source and target. The source is farthest from the target at the center of the spectrum. It is nearest at the extreme ends of the spectrum.

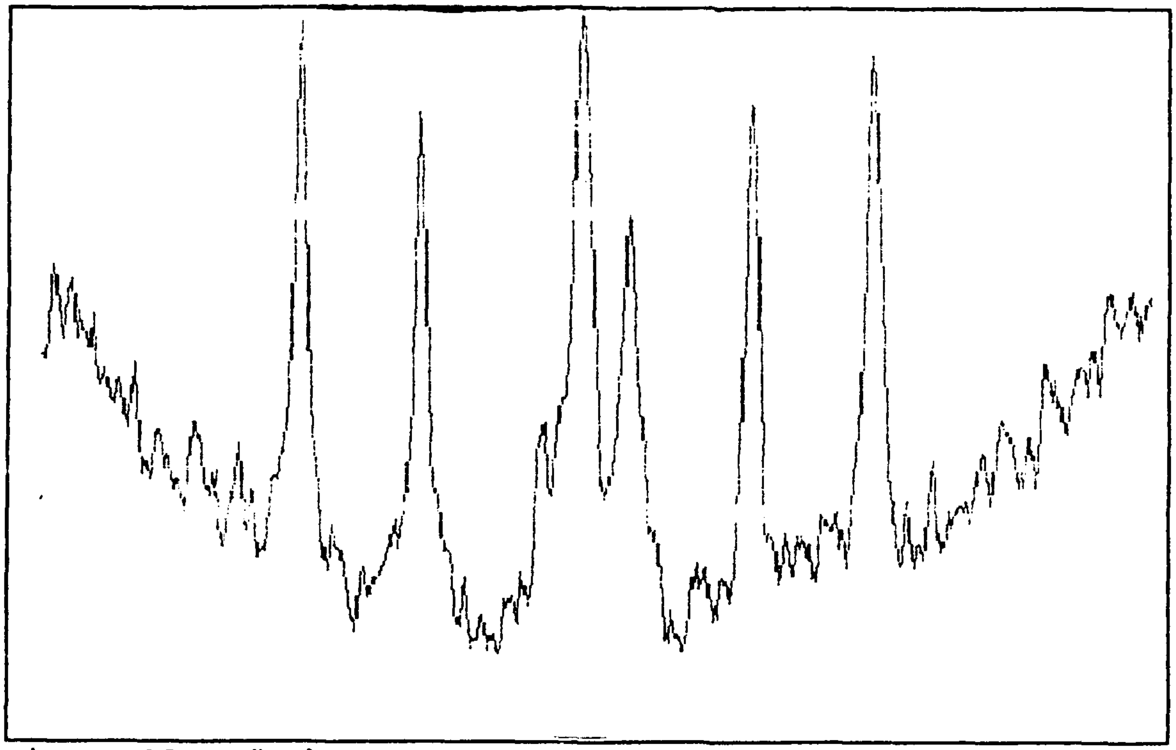

Figure 13 Mössbauer spectrum collected using cem detector system. Collected in 26 days with 2000 Å of ^{57}Fe on stainless steel foil.

For the cem spectrum in Figure 13, counts were accumulated with bias of 2500 volts applied to the cems. This put 2500 volts at the collector and 385 volts at the funnel. A trial run indicated a smaller than expected signal-to-noise ratio. During the trouble shooting phase, described later, the signal

lead from cem number I was disconnected. Data were collected using cems II and III only for 26 days.

Table II Comparison of signal to background ratio, proportional counter and cem systems using a 36.6 mCi ^{57}Co source and 2000 Å of ^{57}Fe on 1 mil stainless steel foil as target.

Peak No.	Height of Peak as Percent of Background: Proportional Counter	Cem System
1	5.5	1.5
2	5.4	1.4
3	2.0	0.5
4	3.4	1.5
5	2.9	1.0
6	4.9	1.5
7	5.5	1.4

The spectra collected by both systems were analyzed by the peak fitting program in the MS-1200 computer. The point of interest is the difference between the height of the peaks and the background which can be expressed as a percentage, as summarized in Table II. The best values obtained in the cem spectrum was 1.5% for peak number 4. This is much lower than the 3.4% for the corresponding peak using the proportional counting system.

Trouble Shooting

In seeking a cause for the poor signal-to-noise ratio several counts were taken on the counter/timer in the NIM bin, with and without a source. The counter timer was connected to

Table III Summary of counts from cem detectors with and without source. Averages are of five counts taken for 100 seconds each, summed then divided by 500 to give counts/second.

	Count Rate (counts/sec)	
	With Source	Without Source
Cems I,II, and III connected.	337 ± 1.9	106 ± 1.4
Cems II and III only connected.	376 ± 4.7	31 ± 1.1

an output from the MS-1200 spectrometer. Results are listed in Table III.

These counts indicate that while the dark count rate is much higher than claimed by the manufacturer, the count rate with a source present is substantially higher than the dark count. But since the signal-to-noise ratio is so poor, many of these counts must be occurring at random rather than being correlated to some velocity of the source. Either ions are being produced somewhere other than in the target and are reaching the cems or one or more of the cems is emitting spurious signals. Discussion with technical representatives at Galileo gave no clues to the problem. Galileo did offer to check the cems performance if we sent them back, but there was not time to do this during the thesis quarter. It was recognized that the shape of the gamma ray beam could affect the background count rate, as well as the signal strength.

To determine the shape of the source beam at the target, the system was brought to atmospheric pressure and the target and detector assemblies were removed. Dental X-ray films were

placed in the position of the target and exposed to the source gamma beam. The clearest picture was obtained with a 30 minute exposure. The picture is shown below in Figure 14 along with a diagram of the cem positions relative to the film.

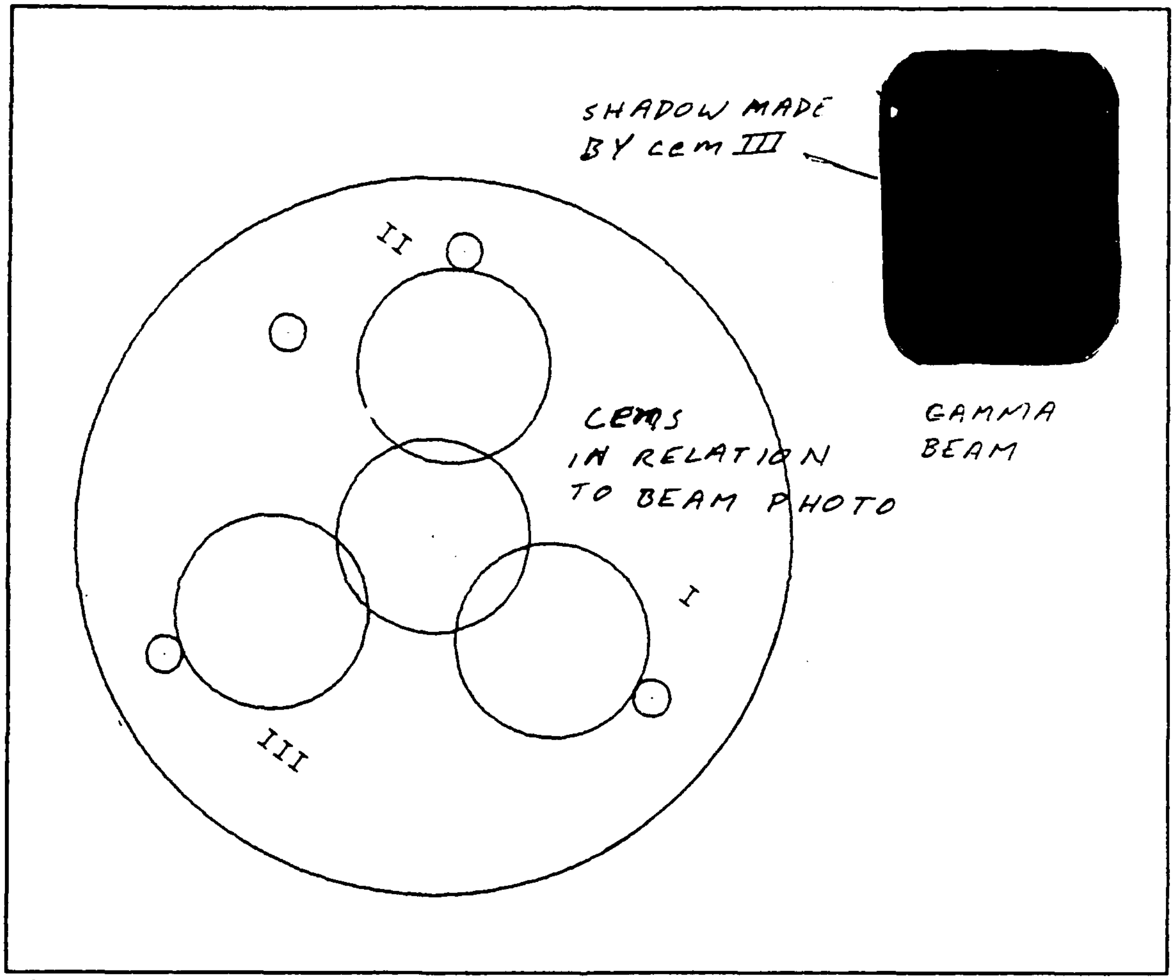

Figure 14 X-ray film exposure of gamma beam.

A shadow produced by cem III is clearly evident in the picture. There is also a shadow cast by cem II which is just barely discernible. The gamma beam is colliding with those cems, more with cem III than cem II. The gamma rays could certainly produce ionizations in the material of the cems thus

causing a large count rate with the source present whether resonant absorptions are taking place in the target or not. However, this does not explain the high dark count rate.

In an attempt to identify the faulty cem the signal lead from cem I was disconnected before reassembling the system. cem I was chosen because it was the easiest to disconnect. Because of time constraints, the system was reassembled before the x-ray film was developed, otherwise one of the cems in the beam path should have been disconnected.

Additional counts were made with only cems II and III providing a signal. Count rate with the source present actually increased slightly. This indicates that cem I was contributing little or nothing to the count rate and is makes sense of the majority of counts are due to electrons liberated directly from the cems by the gamma rays. Dark count rate decreased significantly, from approximately 100 counts per second to 32 counts per second. The reason is not clear unless cem I was the primary source of dark counts but the gamma ray beam striking cems II and III is the source of high background.

V. Conclusions and Recommendations

The goal of this project was partly satisfied. The vacuum chamber reaches and maintains a pressure well within the needs of the detector system. The system is safe and reliable. The output signal from the detectors is compatible with the spectrometry system and spectra can be collected. However, rather than having an improved signal to noise ratio compared to the proportional counting system, the ratio is worse.

It is clear that cems II and III must be moved from the path of the gamma ray beam. This may be difficult to do; even though some adjustment in the radial position of the cems was allowed in the design, they are already close to the wall of the electrical shroud. They should be moved as far back as possible, even if the shroud must be removed.

The shroud may not be a good electric field restraint. The holes made for out-gassing may be large relative to the electric field strength. If so, the field can protrude through the holes and attract ions from the rest of the vacuum chamber to the vicinity of the cems. Attempts to find a fine mesh copper screen to replace the present shroud should continue. As an alternative, screen could be placed directly in front of the cem openings and be subject to a negative bias potential. At the same time, low energy ions could be prevented from entering the cems, reducing the background

count. Such a screen was originally intended as part of this project or as a follow-on.

This system has great potential for use in Mössbauer spectrometry if the high background count problem is solved.

Appendix A: Counts Taken From cems.

Three cems operating, I, II, and III. Five counts taken for 100 seconds each. Source present.

Count No.	Counts per 100 seconds.
1	34110
2	33310
3	33789
4	34068
5	33196

Average, 33694.6, standard deviation, 423.5, standard error of the mean, 189.

Three cems operating, I, II, and III. Five counts taken for 100 seconds each. No source present.

Count No.	Counts per 100 seconds.
1	10097
2	10595
3	10569
4	10900
5	10854

Average, 10603, standard deviation, 319.6, standard error of the mean, 143.

Two cems operating, II and III. Five counts taken for 100 seconds each. Source present.

Count No.	Counts per 100 seconds.
1	39325
2	37888
3	37141
4	36826
5	36888

Average, 37613.6, standard deviation, 1045.7, standard error of the mean, 467.7.

Two cems operating, II and III. Five counts taken for 100 seconds each. No source present.

Count No.	Counts per 100 seconds.
1	3067
2	2991
3	3192
4	3610
5	3237

Average, 3219.4, standard deviation, 239.3, standard error of the mean, 107.0.

Appendix B: Proportional Counter and Cem System Peak Statistics.

File name: C:\DRV91\EF2KA4DC

Region of interest: from channel 20 to channel 1000.

981-Channel Chi Squared is 0.0643260256291 after 3 iterations.

Parameters	Best value	Last delta	Last % Change
1 Height	1354.016357	26.549316	1.961
2 Pos.	260.609528	0.142853	0.055
3 HWHM	6.723160	0.001000	0.000
4 Height	1320.772095	0.000000	0.000
5 Pos.	365.0666[illegible]	[illegible]	-0.000
6 HWHM	5.589531	0.001000	0.000
7 Height	484.284149	-9.883362	-2.041
8 Pos.	468.038116	-0.4[illegible]	-0.092
[illegible]	5.704791	0.001000	0.000
10 Height	829.840149	-16.935486	-2.041
11 Pos.	503.895233	0.428558	0.085
12 HWHM	9.507985	0.001000	0.000
13 Height	714.925903	0.000000	0.000
14 Pos.	545.333313	1.000000	0.183
15 HWHM	7.343062	0.001000	0.000
16 Height	1210.514526	0.000000	0.000
17 Pos.	649.609558	0.142883	0.022
18 HWHM	6.390644	0.001000	0.000
19 Height	1357.336792	-27.700806	-2.041
20 Pos.	752.895264	0.428589	0.057
21 HWHM	5.776761	0.001000	0.000

Peak num.	Position (mm/s)	Height % Bkgd	Relative Height	Half width Half Max	Area	Area % Tot.
1	260.609528	0.054923	0.99762	6.7232	28598.76	18.9789
2	365.066681	0.053642	0.97437	5.5895	23192.80	15.3913
3	468.038116	0.019680	0.35743	5.7048	8679.40	5.7599
4	503.895233	0.033724	0.61258	9.5080	24787.50	16.4496
5	545.333313	0.029053	0.52772	7.3431	16492.56	10.9449
6	649.609558	0.049162	0.89299	6.3906	24303.26	16.1282
7	752.895264	0.055053	1.00000	5.7768	24633.26	16.3472

PROPORTIONAL COUNTER STATISTICS

File name: C:\DRV91\CHCE29JA

Region of interest: from channel 20 to channel 1000.

981-Channel Chi Squared is 0.0641576914644 after 4 iterations.

Parameters	Best value	Last delta	Last % Change
1 Height	5969.446289	-121.825684	-2.041
2 Pos.	260.752380	0.142853	0.055
3 HWHM	5.306942	0.001000	0.000
4 Height	5480.088867	0.000000	0.000
5 Pos.	364.133331	0.142853	0.039
6 HWHM	6.187714	0.001000	0.000
7 Height	1907.127808	0.000000	0.000
8 Pos.	468.066681	-1.000000	-0.214
9 HWHM	5.494209	0.001000	0.000
10 Height	6181.250586	-325.[illegible]	[illegible]
[illegible]	502.466675	-1.000000	-0.199
12 HWHM	[illegible]	[illegible]	[illegible]
13 Height	3973.466797	-209.129883	-5.263
14 Pos.	545.466675	1.000000	0.183
15 HWHM	8.606250	0.001000	0.000
16 Height	5977.863770	229.917969	3.846
17 Pos.	649.466675	0.142883	0.022
18 HWHM	6.009034	0.001000	0.000
19 Height	5544.814453	213.262207	3.846
20 Pos.	753.323792	-0.142883	-0.019
21 HWHM	6.952714	0.001000	0.000

Peak num.	Position (mm/s)	Height % Bkgd	Relative Height	Half width Half Max	Area	Area % Tot.
1	260.752380	0.014375	0.96327	5.3069	99524.09	12.8034
2	364.133331	0.013678	0.88576	6.1877	106528.98	13.7045
3	468.066681	0.004764	0.30850	5.4942	32918.11	4.2348
4	502.466675	0.015442	1.00000	10.1426	196961.31	25.3383
5	545.466675	0.009926	0.64280	8.6062	107431.94	13.8207
6	649.466675	0.014923	0.96639	6.0090	112849.73	14.5177
7	753.323792	0.013821	0.89501	6.9527	121113.13	15.5807

Cem DETECTOR STATISTICS

Appendix C: Equipment List.

Channeltrons, model 4820/EIC/SC.

Channeltron number	I	II	III
Serial number	68355	68165	68166
Factory test bias voltage	2400	2500	2400
Current, microamps	22.2	21.1	22.4
Gain	1.13×10^8	1.02×10^8	1.02×10^8
FWHM	62%	54%	67%

Equipment in NIM bin, all manufactured by EG&G Ortec.

Model 419 Pulse Generator
Model 996 Timer/Counter
Model 521 Single Channel Analyzer
Model 572 Amplifier
Model 659 High Voltage Power Supply and
Model 556 High Voltage Power Supply.

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Vita

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13. ABSTRACT *(Maximum 200 words)*
A channel electron multiplier (cem) detector system was designed and built to provide a signal to a Mossbauer spectroscopy system in an effort to obtain a better signal-to-noise ratio than achievable with a proportional counter system. Three cems are contained in a vacuum chamber built using standard components. Gamma rays of 14.4-keV energy from a Co-57 source enter from outside the chamber through a window and are absorbed by an Fe-57 target near the cems. The target emits internal conversion electrons which are collected by the cems and a Mossbauer spectrometer collects the signal. The system produces a usable signal, but the signal-to-noise ratio is unacceptably high. The strongest peak has a maximum count only 2.1% of the total background count. The corresponding peak in a spectrum collected using the proportional detector system is 3.4% of the total background count. One possible cause is that some gamma rays are striking two of the cems, producing ionizations in the detectors. The cems need to be repositioned. A screen should be added in front of the cems and grounded to reduce counting of low energy electrons. These improvements should reduce the background count rate.

14. SUBJECT TERMS
Mossbauer Spectrometry, channel electron multipliers, detectors, spectrometry, vacuum systems, conversion electronspectrometry

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